JP03200805

Specification

1. Title of the Invention

Method for Purifying Water-Soluble Polyvinyl Acetal

2. Claim

(1) A method for purifying water-soluble polyvinyl acetal characterized by heating an aqueous solution containing water-soluble polyvinyl acetal to form a dense aqueous solution layer of said water-soluble polyvinyl acetal and a water layer and separating the water layer from the dense aqueous solution of polyvinyl acetal.

3. Detailed Description of the Invention

(Applicable Industrial Field)

This invention relates to a method for purifying water-soluble polyvinyl acetal.

(Background Art)

Polyvinyl alcohol and an acrylic resin have heretofore been used as a ceramic binder, but both of them have poor handling property since the former one is hard and fragile and the latter one is too soft. In contrast, a polyvinyl butyral resin has an appropriate hardness, but, since many of conventional polyvinyl butyral resins have a butyralation degree of 50 mol% to 80 mol%, they are not water-soluble and are only soluble to organic solvents.

Accordingly, JP-A-62-156112, for example, proposes a

polyvinyl acetal resin wherein an average chain length of an alkyl group in an acetal ring is C0.34 to C1.70. However, the polyvinyl acetal resin having the short chain length of alkyl group in acetal ring lacks in flexibility.

(Problems that the Invention is to Solve)

The inventors had conducted extensive research in the aim of obtaining a polyvinyl butyral resin that has an appropriate flexibility and is water-soluble to find that it is possible to obtain the water-soluble polyvinyl butyral resin as described above by maintaining a butyralation degree to a predetermined small range by reacting a predetermined amount of butyl aldehyde with an aqueous solution of polyvinyl alcohol.

In general, a purification of a water-soluble resin is performed mainly by employing a re-precipitation method. That is, an organic solvent serving as a poorly soluble agent for a resin is added to an aqueous solution of the resin, and impurities such as ions are dissolved into water to precipitate the resin, thereby separating the resin from the impurities.

However, such method has problems of the risk of using the organic solvent and a post-processing of the water/organic solvent mixture.

Therefore, the inventors had conducted an extensive research in order to solve the above-described problems, and, as a result, found a purification method capable of effectively

eliminating impurities, particularly ions, from an aqueous solution of water-soluble polyvinyl acetal by a treatment in a water system, thereby accomplishing this invention.

That is, an object of this invention is to provide a method for purifying water-soluble polyvinyl acetal.

(Means for Solving the Problems)

A method for purifying water-soluble polyvinyl acetal according to this invention is characterized by heating an aqueous solution containing water-soluble polyvinyl acetal to form a dense aqueous solution layer of said water-soluble polyvinyl acetal and a water layer and separating the water layer from the dense aqueous solution of polyvinyl acetal.

Water-soluble polyvinyl butyral is obtainable by: dissolving polyvinyl alcohol into water; reacting a predetermined amount of butyl aldehyde in the presence of an acid catalyst such as hydrochloric acid, for example; and neutralizing with alkali such as sodium hydroxide, for example.

Therefore, an aqueous solution of thus-obtained water-soluble polyvinyl acetal contains impurities such as chlorine ions and sodium ions.

According to this invention, it is possible to obtain an aqueous solution of high-purity polyvinyl acetal from which the impurities such as the chlorine ions and the sodium ions are eliminated by: heating the aqueous solution containing

such water-soluble polyvinyl acetal, preferably with stirring; separating a dense aqueous solution layer (upper layer) of water-soluble polyvinyl acetal and a water layer (lower layer) from each other; separating the water layer serving as the lower layer by extraction from the water-soluble acetal dense aqueous solution layer of the water-soluble polyvinyl acetal; and repeating such separation operation when so required.

The method of this invention is suitable for purification of an aqueous solution of polyvinyl acetal having a resin concentration of 20 wt% or less. Also, a heating temperature of the aqueous solution may ordinarily be within a range of from 50°C to 100°C, preferably from 80°C to 100°C.

(Effect of the Invention)

As described above, it is possible to effectively eliminate the impurities from the aqueous solution of water-soluble polyvinyl acetal particularly containing inorganic ions as the impurities by this invention. Particularly, according to this invention, it is possible to obtain an aqueous solution of purified polyvinyl acetal by producing water-soluble polyvinyl acetal in a water system and eliminating the impurities by a treatment in the water system.

The purified polyvinyl acetal aqueous solution that does not contain the impurity ions is suitably used as an aqueous binder for ceramic green sheet for ceramic condenser

production in electronics industry, for example. Also, the purified polyvinyl acetal aqueous solution is usable as a coating agent for aqueous ink jet recording papers, a hydrophilic property imparting additive, and the like.

(Examples)

Hereinafter, this invention will be described specifically in conjunction with examples.

Example 1

790 parts by weight of polyvinyl alcohol having a polymerization degree of 500 and a saponification degree of 88 mol% was added to 2120 parts by weight of a pure water and dissolved with stirring at 90°C for 2 hours. After that, the solution was cooled to 45°C, and 27 parts by weight of 35 wt% chloric acid was added thereto, followed by cooling to 35°C. Subsequently, 62 parts by weight of butyl aldehyde having a purity of 99 wt% was dripped into the solution during 2 hours, followed by a reaction at 35°C for 3 hours, thereby obtaining a white turbid solution.

66 parts by weight of a 10 wt% sodium hydroxide aqueous solution was added to the white turbid solution with stirring, followed by neutralization and cooling to 20°C, thereby obtaining a transparent solution.

5 parts by weight of the solution was dispensed on an aluminum plate, followed by vacuum drying at 70°C for 3 hours, and it was confirmed that the solution contained a solid matter

of about 25 wt% and was a polyvinyl butyral resin from its infrared absorption spectrum. A butyralation degree of the resin measured by a method of JIS K 6728 was about 10 mol%.

Also, a sodium ion concentration in the solution was detected to be 2000 ppm as a result of measurement by an atomic absorption device, and a chlorine ion concentration measured by ion chromatography was about 3000 ppm.

500 parts by weight of the solution was added to 4500 of a pure water to achieve a total amount of 5000 parts by weight, followed by heating to 95°C with stirring, whereby the solution was separated into an upper layer that was opaque and viscous and a lower layer that was transparent. The lower layer was separated at a high temperature to obtain about 500 parts by weight of the viscous upper layer. This layer became a transparent solution when cooled to 20°C.

A content of a solid matter in the solution that was measured in the same manner as described above was 23 wt%, and the solid matter was a polyvinyl butyral resin having a butyralation degree of about 10 mol%. It was confirmed that a sodium ion concentration and a chlorine ion concentration in the solution were reduced to about 200 ppm and about 310 ppm.

Example 2

255 parts by weight of polyvinyl alcohol having a polymerization degree of 1000 and a saponification degree of

98.5 mol% was added to 2700 parts by weight of a pure water and dissolved with stirring at 90°C for 2 hours. After that, the solution was cooled to 45°C, and 20 parts by weight of 35 wt% chloric acid was added thereto, followed by cooling to 35°C. Subsequently, 16 parts by weight of butyl aldehyde having a purity of 99 wt% was dripped into the solution during 2 hours, followed by a reaction at 35°C for 3 hours, thereby obtaining a white turbid solution.

66 parts by weight of a 10 wt% sodium hydroxide aqueous solution was added to the white turbid solution with stirring, followed by neutralization and cooling to 20°C, thereby obtaining a transparent solution.

It was confirmed in the same manner as in Example 1 that the solution contained about 8 wt% of a polyvinyl butyral resin having a butyralation degree of about 8 mol%. Also, a sodium ion concentration in the solution was 1500 ppm, and a chlorine ion concentration was 2200 ppm.

500 parts by weight of the above solution was added to 4500 of a pure water to achieve a total amount of 5000 parts by weight, followed by heating to 95°C with stirring, whereby the solution was separated into an upper layer that was opaque and viscous and a lower layer that was transparent. The lower layer was separated at a high temperature to obtain about 200 parts by weight of the viscous upper layer. This layer became a transparent solution when cooled to 20°C.

A content of a solid matter in the solution that was measured in the same manner as described above was about 21 wt%, and the solid matter was a polyvinyl butyral resin having a butyralation degree of about 8 mol%. It was confirmed that a sodium ion concentration and a chlorine ion concentration in the solution were reduced to about 60 ppm and about 90 ppm.